



Sir:

I, Hisayuki Kuwahara, declare as follows:

**I. IDENTIFICATION OF DECLARANT**

I am employed by Mitsubishi Gas Chemical Company, INC and hold the position of research manager of Hiratsuka research laboratory.

My educational background is as follows:

Graduated from Osaka University

Master's Degree in Chemistry

**II. DETAILS OF EXPERIMENTS**

I have conducted personally or under my direction and control the following experiments:

**1. Purpose of Experiments**

The purpose of the following experiments is to compare the low-temperature curability of epoxy resin curing agents using Amino Compound A in Example 1 of U.S. 6,562,934 and Amino Compound B in Example 2 of U.S. 6,562,934 added with salicylic acid and that of the epoxy resin curing agent of the present invention.

**2. Preparation of epoxy resin curing agents**

(1) 19g of Amino Compound A and 1.0g of salicylic acid were weighed and charged to a sample bottle with the volume of 50 ml. Then, the mixture was stirred at temperature of 80°C to solve the components to obtain a curing agent "J" as a homogeneous solution.

(2) The Amino Compound A was also used as a curing agent without adding salicylic acid (= a curing agent "K").

(3) In the same manner as above, 10g of Amino Compound B and

1.0g of salicylic acid were weighed and charged to a sample bottle with the volume of 50 ml. Then, the mixture was stirred at temperature of 80°C to solve the components to obtain a curing agent "L" as a homogeneous solution.

(4) The Amino Compound B was also used as a curing agent without adding salicylic acid (= a curing agent "M").

### 3. Evaluation

The epoxy resin curing agents "J", "K", "L", and "M" were mixed respectively with bisphenol A type liquid epoxy resin with an epoxy equivalent weight of 216g/eq, manufactured by Japan Epoxy Resins Co., Ltd., brand name; Epikote 801, at a ratio shown in Table 1 and 2 to obtain epoxy resin compositions.

The epoxy resin compositions thus obtained were cured under the conditions of 5°C and 80% RH to prepare epoxy resin cured coating films. The properties of the epoxy resin cured coating films were evaluated and the results were shown in Table 1 and 2.

Table 1

	Reference Example 1	Reference Example 2
Epoxy resin composition (g)		
Epikote 801	100	100
Epoxy Resin Curing Agent J	39	
Epoxy Resin Curing Agent K		37
Property of a cured coating film		
Appearance		
Gloss	◎	◎
Clarity	◎	◎
Leveling	△	△
Dryness (16 hours/1day/4days/7days)	x /◎/◎/◎	x /○/◎/○
Water resistance (16 hours/1day/4days/7days)	x /△/◎/◎	x /△/◎/○

Table 2

	Reference Example 3	Reference Example 4
<b>Epoxy resin composition (g)</b>		
Epikote 801	100	100
Epoxy Resin Curing Agent L	84	
Epoxy Resin Curing Agent M		80
<b>Property of a cured coating film</b>		
<b>Appearance</b>		
Gloss	◎	◎
Clarity	◎	◎
Leveling	◎	◎
<b>Dryness</b>		
(16 hours/1day/4days/7days)	✗ / ✗ / Δ / ◎	✗ / ✗ / Δ / ◎
<b>Water resistance</b>		
(16 hours/1day/4days/7days)	✗ / ✗ / Δ / ◎	✗ / ✗ / Δ / ◎

For purpose of comparison, Example 1 and Comparative Example 1 of the present application are shown in the following Table 3:

Table 3

	Example 1	Comparative Example 1
<b>Epoxy resin composition (g)</b>		
Epikote 801	100	100
Epoxy Resin Curing Agent A	50	
Epoxy Resin Curing Agent C		48
<b>Property of a cured coating film</b>		
<b>Appearance</b>		
Gloss	◎	◎
Clarity	◎	◎
Leveling	◎	◎
<b>Dryness</b>		
(16 hours/1day/4days/7days)	Δ / ◎ / ◎ / ◎	✗ / ✗ / ◎ / ◎
<b>Water resistance</b>		
(16 hours/1day/4days/7days)	Δ / O / ◎ / ◎	✗ / Δ / ◎ / ◎

#### 4. Result

The result of the experiments shown in Table 1 and 2 indicates that, in the case of a curing agent using Amino Compound A and B of U.S. 6,562,934 respectively as a diamine component, the low-temperature curability is lower than the curing agent of the present invention. In addition, even if salicylic acid is added with the curing agent using Amino

Compound A and B of U.S. 6,562,934, any improvement in low-temperature curability cannot be achieved. More precisely, in the case of a curing agent using Amino Compound A, addition of salicylic acid does not bring any improvement in water resistance. Moreover, in the case of a curing agent using Amino Compound B, addition of salicylic acid does not bring any improvement in dryness and in water resistance.

On the other hand, in the case of the curing agent of the present invention, addition of salicylic acid brings improvement both in dryness and in water resistance.

Though the reasons for these results are not clear actually, it can be assumed that these results may be caused by the following reasons:

(1) Amino Compound A in Example 1 of U.S. 6,562,934 comprises more than 2% by weight of unreacted polyamine which may bring deterioration in the activity of salicylic acid.

(2) Amino Compound B in Example 2 of U.S. 6,562,934 is obtained by addition reaction of MXDA and styrene with the reaction molar ratio of 1:2, which brings decrease of proportion of 1:1 adduct in the composition of the resulting curing agent and may bring deterioration in the activity of salicylic acid.

### III. CONCLUSION

The foregoing experiments demonstrate that, in the case of a curing agent using Amino Compound A and B of U.S. 6,562,934 as a polyamine component, addition of a curing accelerator such as salicylic acid does not bring any improvement in curability under the condition of low temperature.

That is, the epoxy resin curing agent according to the claimed invention comprising a polyamino compound obtained by addition reaction of aliphatic diamine represented by the formula (1) and styrene with the reaction molar ratio of 1:1 and a curing accelerator comprising an organic compound having at least one carboxyl group and at least one hydroxyl group within the molecule, wherein said polyamino compound contains less than 2% by weight of unreacted aliphatic diamine

represented by the formula (1) achieves an unexpectedly superior low-temperature curability compared to an epoxy resin curing agent according to the prior art.



wherein A is a phenylene group or a cyclohexylene group

#### IV. VERIFICATION CLAUSE

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: Sep. 19, 2007 Signature: H. Burkhardt